Non- Chromium Conversion Coatings on Aluminum

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OH,USA

Annual Report

Special Project No. SPC-98-4005

USAF contract No. F61775-98-WE 054

January, 1999

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	30 January 1999	Final Report

4. TITLE AND SUBTITLE

Non-Chromium Conversion Coatings on Aluminum

5. FUNDING NUMBERS

F61775-98-WE054

6. AUTHOR(S)

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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Technion Israel Institute of Science and Technology,
Technion City
Haifa 32 000
Israel

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES

EOARD
PSC 802 Box 14
FPO 09499-0200

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

SPC-98-4005

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT	12b. DISTRIBUTION CODE
Approved for public release; distribution is unlimited	A

ABSTRACT (Maximum 200 words)

This report results from a contracted tasking Technion – Israel Institute of Science and Technology as follows: The contractor will investigate non-chromium conversion coatings on aluminum. Conversion coatings are an important corrosion protector and allow for better adhesion of paint and other coatings. Finding an enviromentally sound replacement to current conversion coatings is a high priority USAF goal.

14. SUBJECT TERMS	15. NUMBER OF PAGES
EOARD, Coatings, Fluids, Lubrication, Materials	42
	16. PRICE CODE
	N/A

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19, SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

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1. Introduction

This work was started in 1996 with the aim to develop alternatives for hard chromium coatings and chromate passivation of Al 2024 T3. The results obtained in 1998 are reported in the present report.

In the previous stages of the investigation the deposition from a zinc/phosphate solution under an external cathodic polarization was proposed as a replacement for chromate passivation of Al 2024 T3. An optimal composition of the electrolyte and deposition conditions were determined for small samples (2 x 4 cm^2). Samples with a new protective layer withstood more than 300 hour salt spray test [1].

In the reported period the work continued with relatively large samples (10x10 cm^2). The results showed nonuniformity of the coating thickness over the sample area. Coating nonuniformity took place not only because of an edge effect, but also in the sample center. The thickness nonuniformity could lead to an insufficient corrosion resistance and poor paintability of samples. In order to improve the uniformity, the appropriate pretreatment of the surface presumably is necessary.

In the reported period the composition, structure, corrosion resistance and paintability of zinc/phosphate coatings on Al 2024 T3 were investigated as a function of pretreatment conditions.

2. Experimental

The procedure of the sample preparation included degreasing in the solution described in Table 1, pretreatment and phosphating under an external polarization at a cathodic current density of 1 A/dm^2. The composition of a phosphating electrolyte is shown in Table2. Phosphating was carried out at pH 2.00-2.20. In order to maintain a pH value in the desired range , the correction by H_3PO_4 was used. The electrolyte temperature was 60°C. One ZnAg anode was used for the deposition.

Pretreatment procedures included sand blasting or/and etching in alkaline or acid solutions. In some experiments an etching was followed by the removal of etching products from the surface. Some of experiments were carried out with a pretreatment in a 3 % colloidal SiO_2 solution (Bonder) for the formation of crystallization centers on the sample

surface. The corrosion resistance of zinc / phosphate coatings was estimated according to ASTM B117. Corrosion tests were carried out in Israel Institute of Metals.

In order to examine the paintability of samples, they were coated with an epoxy primer according to IAI PS 24.35, aged for two weeks and then subjected to adhesion tests (Wet Tape Test, Dry Tape Test and Scribed Tape Test) carried out according to standard IAI PS 83.01. A paintability test was carried out in Israel Aircraft Industry, Division 4485. The description of the paintability test is shown in Appendix A. The structure and composition of zinc / phosphate coatings were investigated by SEM, EDS and XRD analyses.

Table 1: Composition of degreaser.

Component	Concentration, % (w.c.)
Sodium silicate	3
Sodium phosphate	2.5
Sodium hydrogen phosphate	1.2
Soap	1

Table 2: Composition of phosphating electrolyte

Component	Concentration
$ZnCO_3$	6 g/l
H_3PO_4	8 ml/l
HNO_3	4 ml/l

3. Results.

3.1. Pretreatment in phosphoric acid.

The immersion of Al 2024 T3 samples in 10-30 % (v.c.) solutions of H_3PO_4 resulted in an intensive etching of the material surface and was accompanied by vigorous evolution of hydrogen. The intensity of the etching increased with increasing H_3PO_4 concentration and solution temperature. After this treatment the surface of Al 2024 T3 samples was covered with a thin dark powdery film which could be removed mechanically by finger or tweezers touch, but not by rinsing in water.

When the temperature of a H_3PO_4 solution and treatment duration were 25° C and 3 min, respectively, the film was hardly detectable under observation with a naked eye. The increase of the solution temperature and treatment duration led to the formation of a well defined film. The Al 2024 T3 surface after the treatment in H_3PO_4 is shown in Figs. 1,2. Separate corroded zones with a size of 5-10 μm could be seen on the surface. These zones seemed to be protrusions or particles surrounded by recessions (white and dark areas, respectively, signed 1 and 2 in Fig. 2). The composition of white areas (1 in Fig.2) included O and Cu in concentration of 27 and 21 % , respectively, as well as Mn and Fe in perceptible concentrations (about 2 %), the remainder being Al. The copper concentration in recessions (dark areas) was equal to about 8 %. The composition of the areas free of the corroded zones (area 3 in Fig.2) was as follows, %: O - 16.37; Mg - 1.57; Al - 79.17; Cu - 2.89. A dark powdery film on the Al 2024 T3 surface formed during etching apparently included the described zones.

As could be seen from the data in Table 3 the increase of the duration of the treatment in a H_3PO_4 solution did not influence noticeably the corrosion resistance of samples. The samples pretreated at 40°C exhibited the same corrosion resistance as those pretreated at 25°C. Samples 269-279 and 281 withstood 240 hour salt spray test. The duration of a salt spray test for samples 269-272 was increased to 336 hours; no corrosion was revealed in the center of the samples. Two-four stains were revealed at the edges of the samples.The microscopic investigation of the coating structure did not allow to reveal a substantial effect of the pretreatment . However, according to visual observation the increase of an

etching temperature and duration resulted in some improvement of the uniformity in a coating thickness across the sample surface.

The unsatisfactory quality of samples 286-292 was found to be associated with the changes in the composition of a phosphating solution during operation [2].

Table 3: Effect of pretreatment in H_3PO_4 solutions on corrosion resistance of Zinc Phosphate coatings.

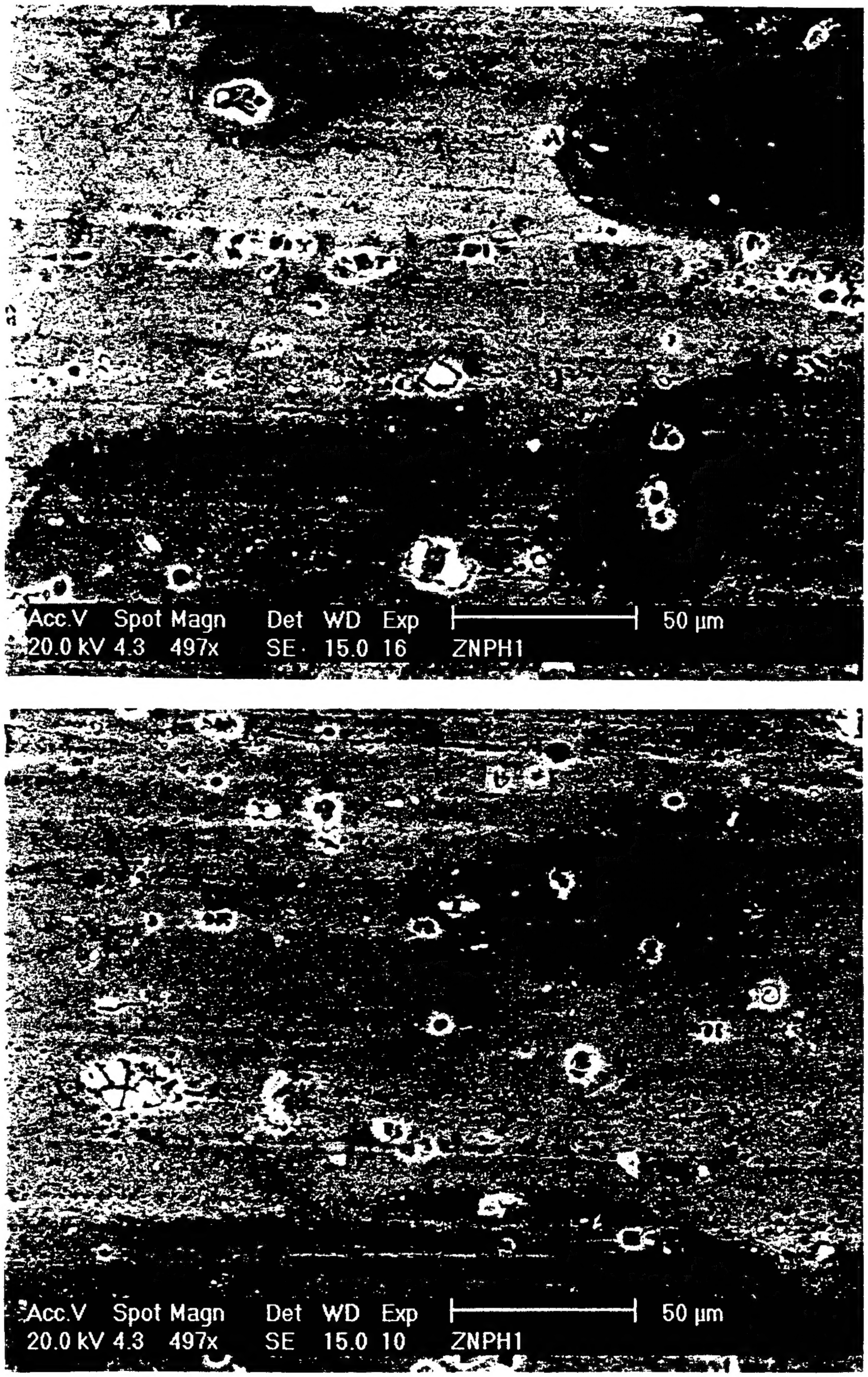
Sample number	Pretreatment procedure H_3PO_4 %(v.c.)	T, °C	D, min	Corrosion resistance (240 h salt spray test)
269		25	17	
270		40	3	
271	30	40	6	
272		40	3	
274		40	6	No corrosion
275	10	25	3	
276		25	6	
278		40	3	
279		40	3	
280 *		40	3	-
281	30	40	3	A few stains appeared along edges
282 *		40	3	-
283 *		40	3	-
284 *		40	6	-
285 *		40	6	-
286		40	6	Black area in the center of the sample was formed after 60 hrs exposure in salt spray. A lot of white corrosion stains appeared in this area after 240 hrs exposure.
287 *		40	6	
288	30	40	6	Nonuniform coatings with stains and uncovered areas. They were not subjected to corrosion test.
289 *		40	6	
290 *		40	3	Samples 288, 291 and 292 were absolutely unsatisfactory.
291		40	6	
292		40	6	

Note: * - Samples were subjected to paintability test.

Samples 280, 282- 285, 287, 289, 290 were subjected to the paintability test. Wet Tape Test showed good adhesion of an epoxy primer to the surface of a zinc/phosphate coating. Along with this, adhesion of a zinc/ phosphate coating to the surface of Al 2024 T3 was found to be unsatisfactory according to the results of Dry tape Test and Scribed Tape Test. The optical microphotographs of tested areas on different samples after Dry Tape Test and Scribed Tape Test are shown in Figs. 4,5. No difference in the structure of the adhesion failure for the samples treated for 3 and 6 min was revealed. The areas of a well adherent coating were randomly distributed over the sample surface. The banded rolling structure of Al 2024 T3 was well seen at stripped areas.

The size and location of the coating adherent areas did not depend on the treatment time. No definite orientation of these areas relative to the direction of rolling bands on the Al 2024 T3 surface was observed. In some cases they were oriented along bands (samples 283,285) and in other cases at an angle to bands (samples 280, 282, 284, 287, 289, 290). The localization of adherent areas was more likely associated with the direction of Tape peeling during Scribed Tape Test than with the morphology of the Al 2024 T3 surface.

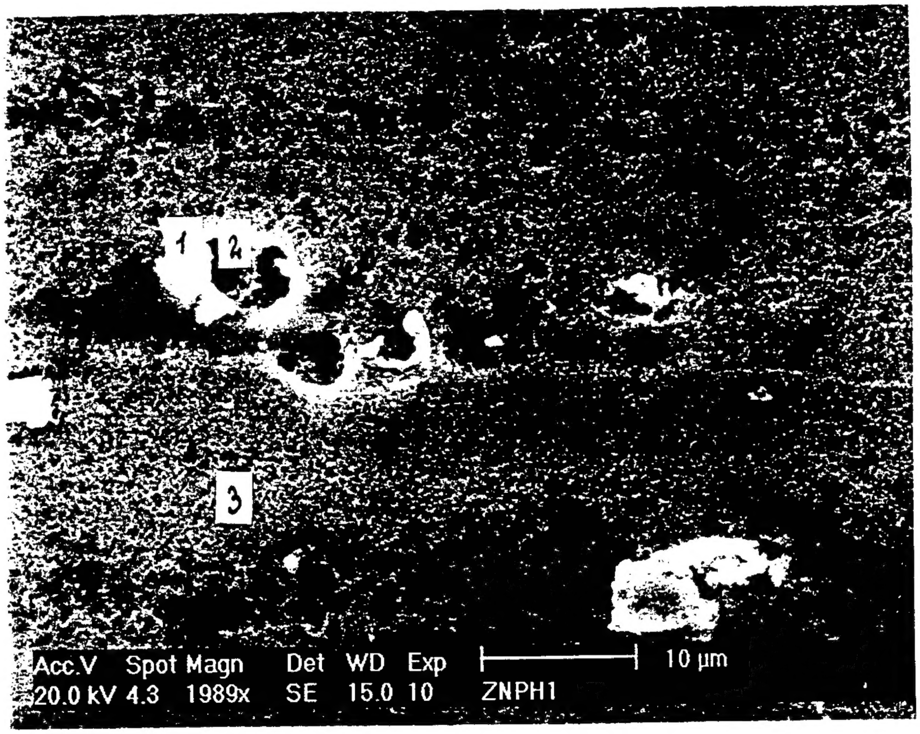
The SEM examination of an adhesion failure structure allowed to distinct the areas of different compositions on the surface of the sample being tested. (Fig.6). The surface composition of dark gray areas (signed 1) corresponds to the composition of Al 2024 T3. The EDS spectrum of the surface in light gray and white areas (signed 2 and 3 in Fig.6, respectively) are shown in Figs 7 and 8, respectively. The composition, atomic concentration (%) in a light gray area was found to be as follows, C - 12.31; O - 53.42; Al - 0.66; P - 13.25; Zn - 20.35. Light gray areas were identified as areas of an zinc / phosphate coating. In these areas the cohesion strength of a zinc / phosphate coating was lower than the adhesion of the coating to an Al 2024 T3 surface. White areas were characterized by a high carbon concentration (about 70 %) and by the presence of Si and Al. These areas corresponded to pieces of an epoxy primer. The examination of surface morphology of adhesion failure at magnification of 2000 showed the presence of the 2-20 μm areas of the zinc / phosphate coating which were scattered among stripped areas (signed 1 and 2 in Fg. 9, respectively). On the other hand no corrosion zones with an increased copper content, which were formed during pretreatment in H_3PO_4, were revealed. It could be suggested that these zones were covered with the zinc / phosphate coating and were responsible for the



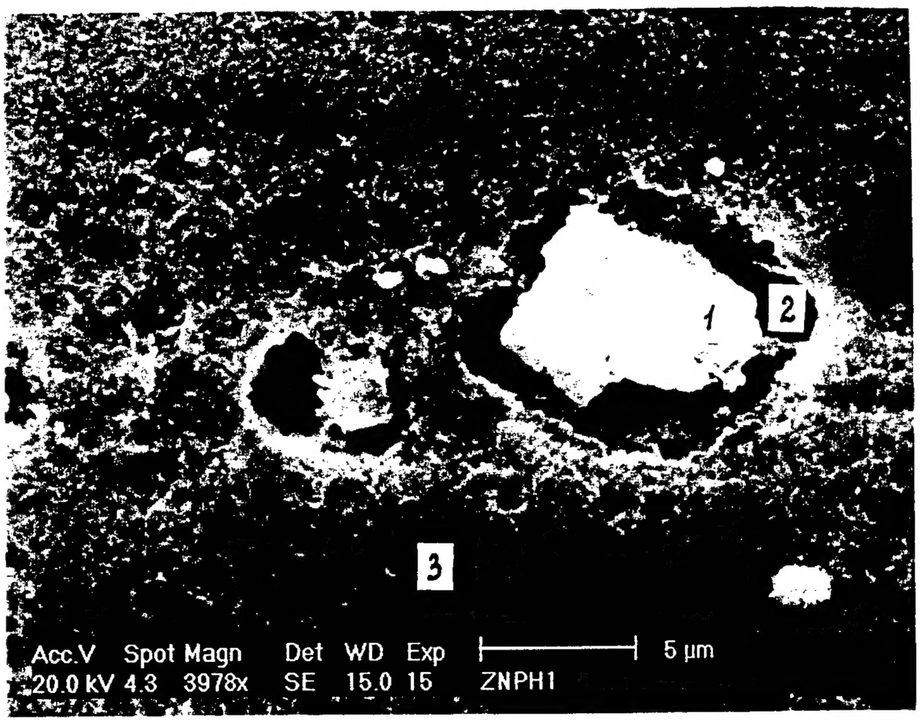
a

b

Fig. 1. Structure of Al 2024 T3 surface after treatment in 30 % H_3PO_4 at 40°C for 10 min. (a) and (b) show two different areas.



a



b

Fig.2. Structure of Al 2024 T3 surface after treatment in 30 % H_3PO_4 at 40°C for 10 min. (a) and (b) show two different areas.

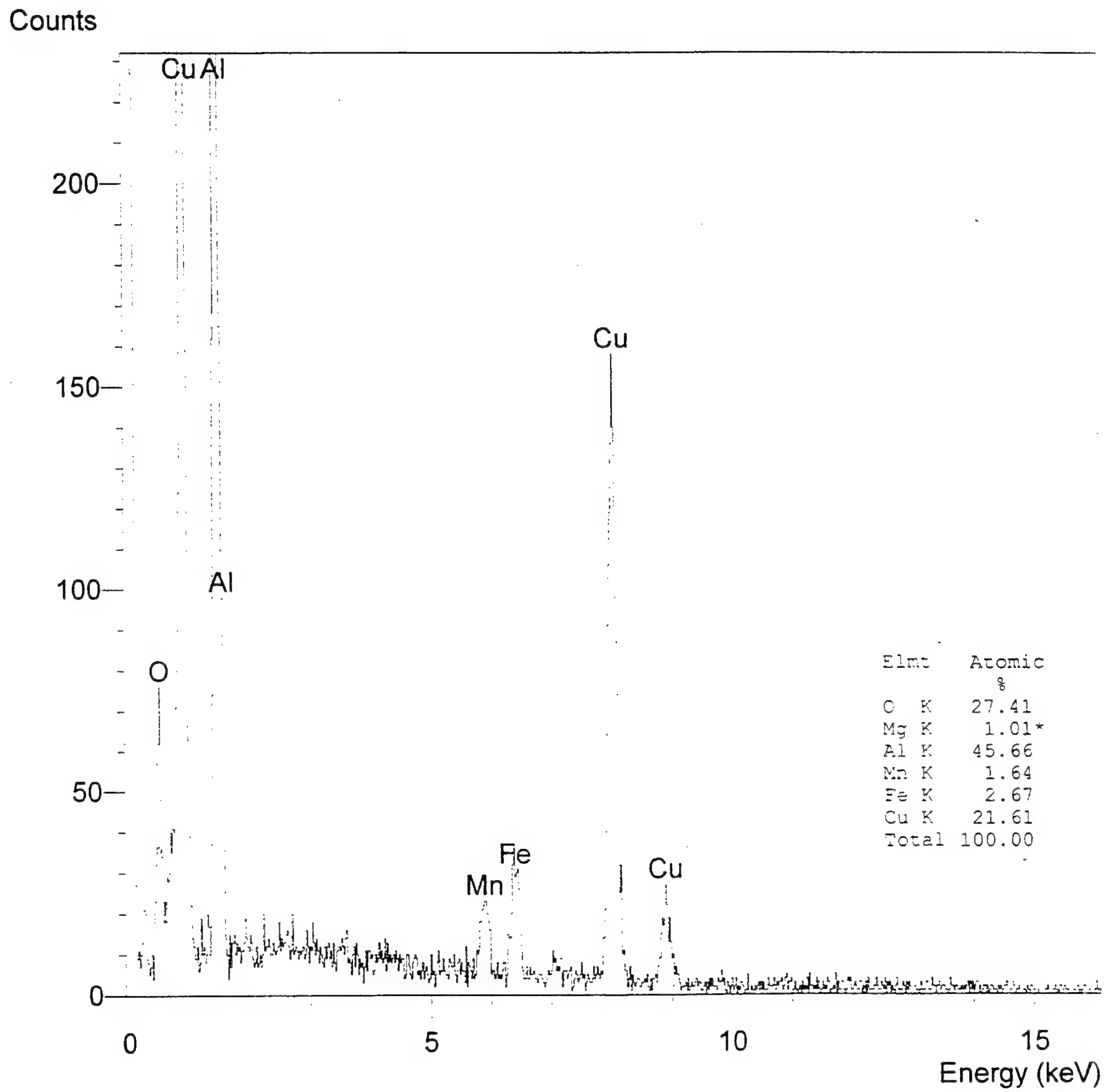
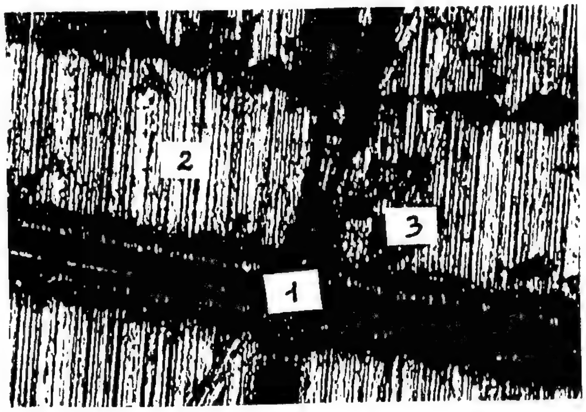
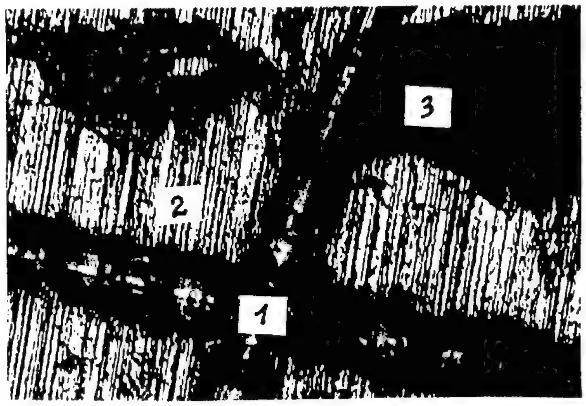


Fig.3. EDS spectrum and composition of particle on Al 2024 T3 surface (signed 1 in Fig.2) after treatment in 30 % H_3PO_4 at 40°C for 10 min.

Sample 280



Sample 282



Sample 283

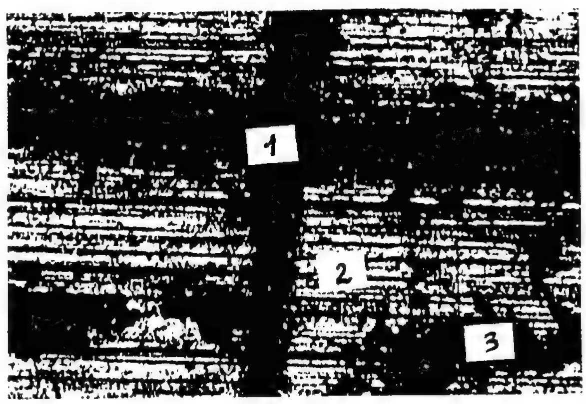
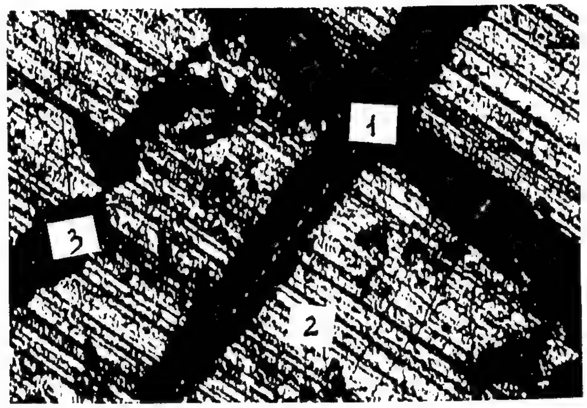
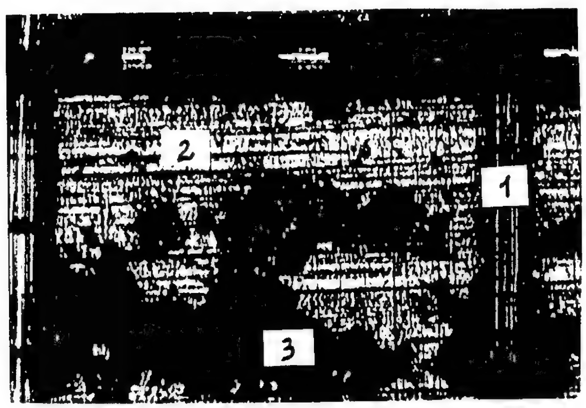


Fig.4. Structure of tested area after Dry Tape Test + Scribed Tape Test. Pretreatment in 30 % H_3PO_4 at 40°C for 3 min. Optical microphotographs. Magnification 50. 1- scratch produced in Scribed Tape Test, 2- stripped area, 3- coating area.

Sample 284



Sample 287



Sample 290

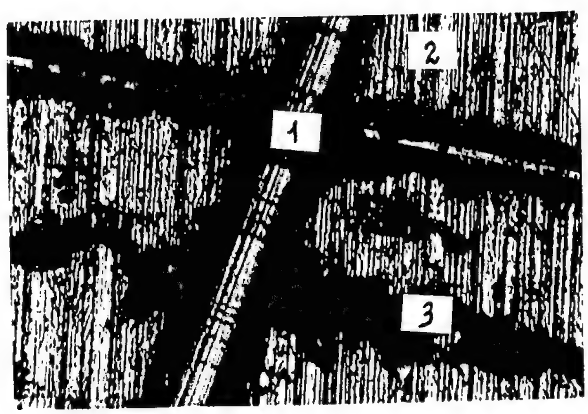


Fig.5. Structure of tested area after Dry Tape Test + Scribed Tape Test. Pretreatment in 30 % H_3PO_4 at 40°C for 6 min. Optical microphotographs. Magnification 50. Magnification 50. 1- scratch produced in Scribed Tape Test, 2- stripped area, 3- coating area.

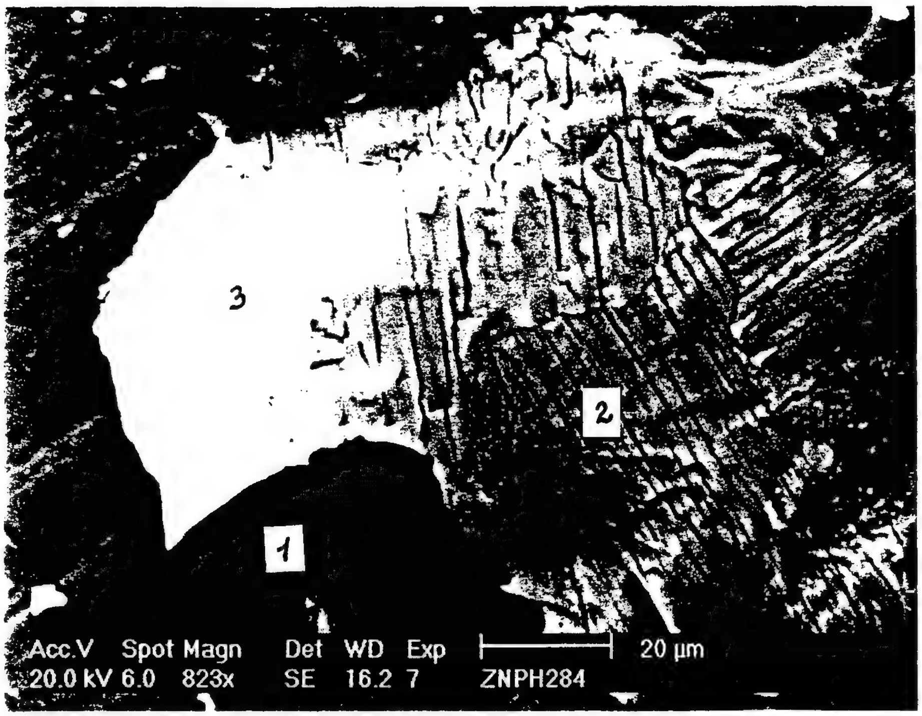


Fig. 6. Surface morphology at area of adhesion failure. Sample 284.

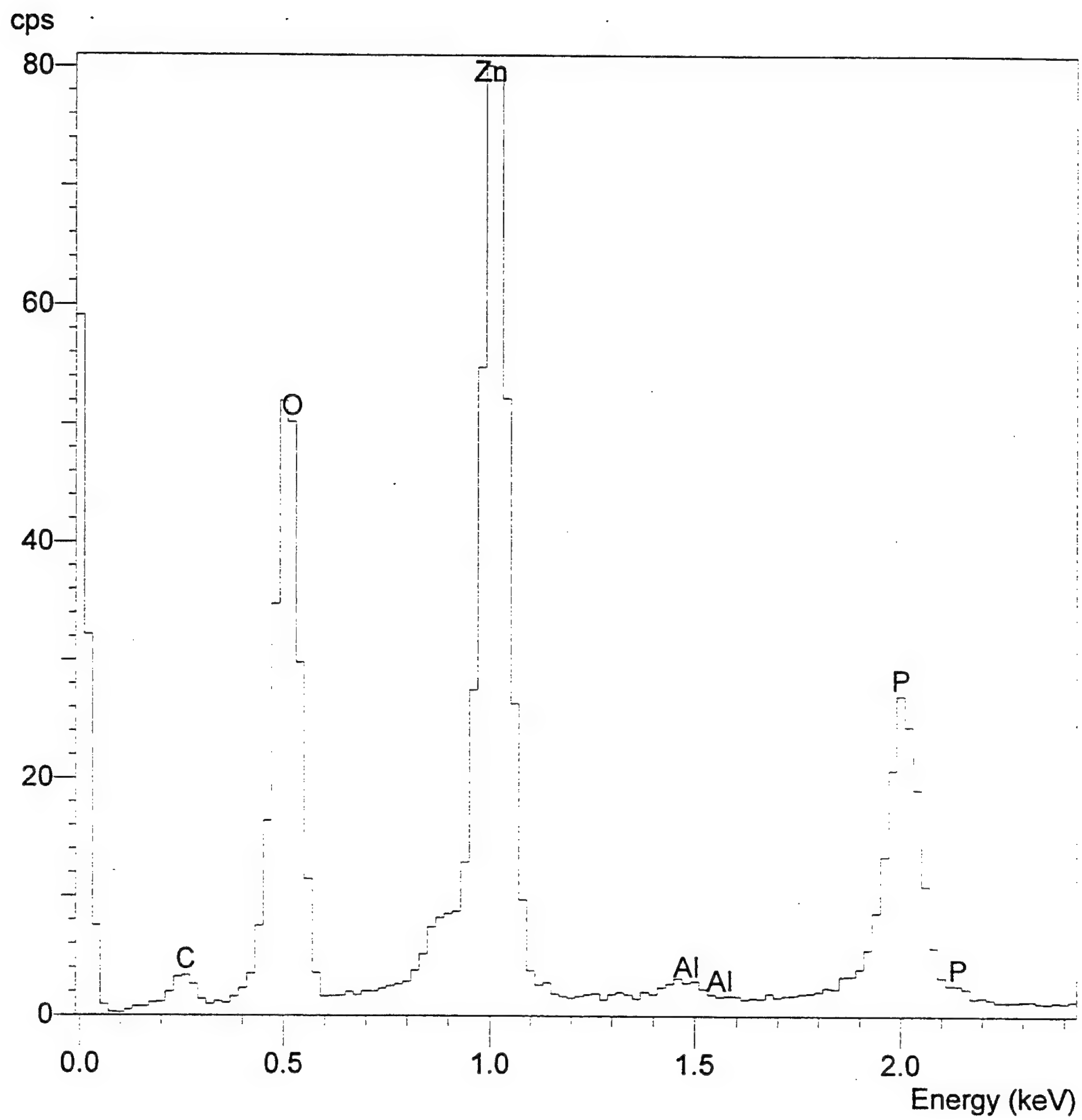


Fig. 7. EDS spectrum of a light gray area (sighed 2 Fig. 6).

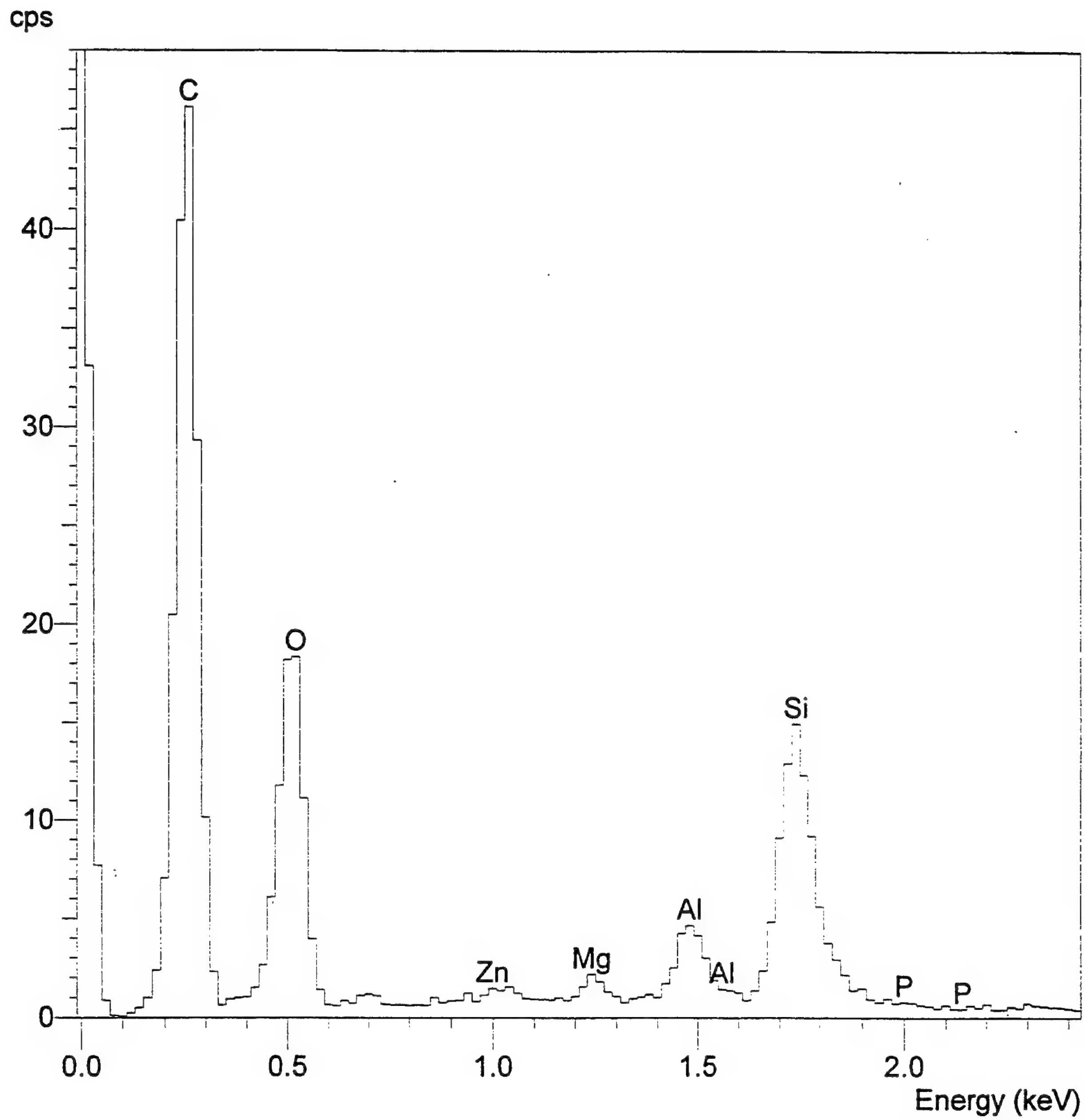
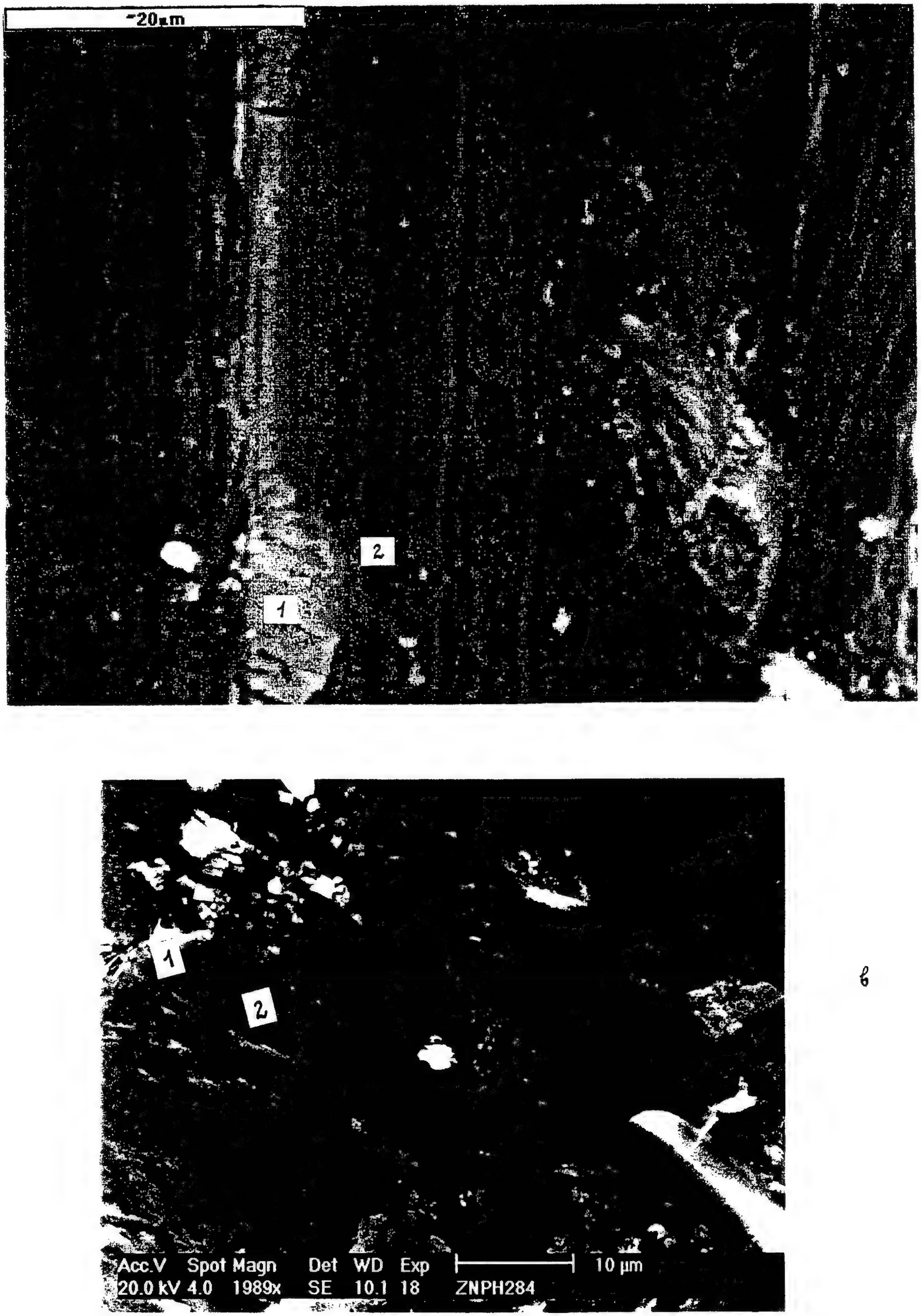


Fig. 8. EDS spectrum of a white area (sighed 3 in Fig. 6).



a

б

Fig. 9. Surface morphology at area of adhesion failure. a - Sample 282. b - Sample 284.

adhesion of the coating to the material surface. A dark powdery film on an etched Al 2024 T3 surface presumably comprised copper enriched protrusions and particles. Protrusions did not lose links with the surface and were apparently responsible for the adhesion of the coating. Particles formed from protrusions as a result of deep etching lost the links with the surface. These unadherent particles could contribute to the decrease of adhesion and could be the reason of the unsatisfactory results of Dry Tape Test and Scribed Tape Test. In order to investigate the effect of the powdery film on corrosion resistance and paintability, a series of experiments with a two stage pretreatment procedure was carried out. The samples of Al 2024 T3 were etched in 30 % (vol) H_3PO_4 at 40°C at the first stage of the pretreatment procedure and after rinsing in water immersed in different acid solutions at the second stage. The conditions and results of these experiments are shown in Table 4. The second stage of the pretreatment was intended to remove the unadherent powdery film formed at the first stage.

The increase of the duration of a H_3PO_4 treatment led to the increase of the temperature and time of the immersion in HNO_3 necessary for the removal of the powdery film from the surface. The additional treatment in the solution of HNO_3 decreased corrosion resistance.The addition of H_2O_2 to a HNO_3 solution accelerated the removal of the film drastically. As can be seen from Table 4 ,the mechanical or chemical (with HNO_3) removal of the powdery film lowered the adhesion of the zinc / phosphate coating to the Al 2024 T3 surface.

3.2. Pretreatment in acidic solutions of H_2O_2.

The composition of acidic solutions of H_2O_2 used for the pretreatment of Al 2024 T as well as the corrosion resistance and paintability of coated samples are shown in Table 5.

The pretreatment in the solution on the base of H_3PO_4 resulted in the formation of a dark powdery film. Therefore the samples 329, 343, 333, 336 were subjected to the second stage of the pretreatment - immersion in the solution containing 100 ml/l HNO_3 and 25 ml/l H_2O_2 at 25°C for 15-20 sec. After this two stage pretreatment no powdery film was revealed on the surface. The structure of the surface is shown in Fig. 10.

Table. 4. Effect of two stage pretreatment on the quality of a treated surface, corrosion resistance and paintability of zinc/ phosphate coatings.

No.	Pretreatment				Powdery film	Corrosion resistance (312 h salt spray test)	Paintability (Dry Tape Test+ Scribed Tape Test)
	1-st stage	2-nd stage					
	D, min	Sol. comp.	T, oC	D, min			
303	6	-	-	-	Present		Did not meet the requirements but on the verge
304						No corrosion	
308	6	-	-	-	Present		Did not meet the requirements but on the verge
315						No corrosion	
317	6		25	3	Present*		Did not meet the requirements
318	12				Present		did not meet the requirements but on the verge
A	15		25	20	Present		
B	15	100ml/l HNO_3	35	17	Removed		Did not meet the requirements
320	10		40	6	Present	Many corrosion stains	
321	5		40	1.5	Removed		Did not meet the requirements
322	4		35	1.5	Removed		Did not meet the requirements
328	5	100ml/l HNO_3 25 ml/l H_2O_2	30	0.3	Removed		Did not meet the requirements but on the verge

Notes:

1) Samples 308 and 315 were treated in H_3PO_4 at 25°C.

2) * The film was mechanically removed from the sample surface.

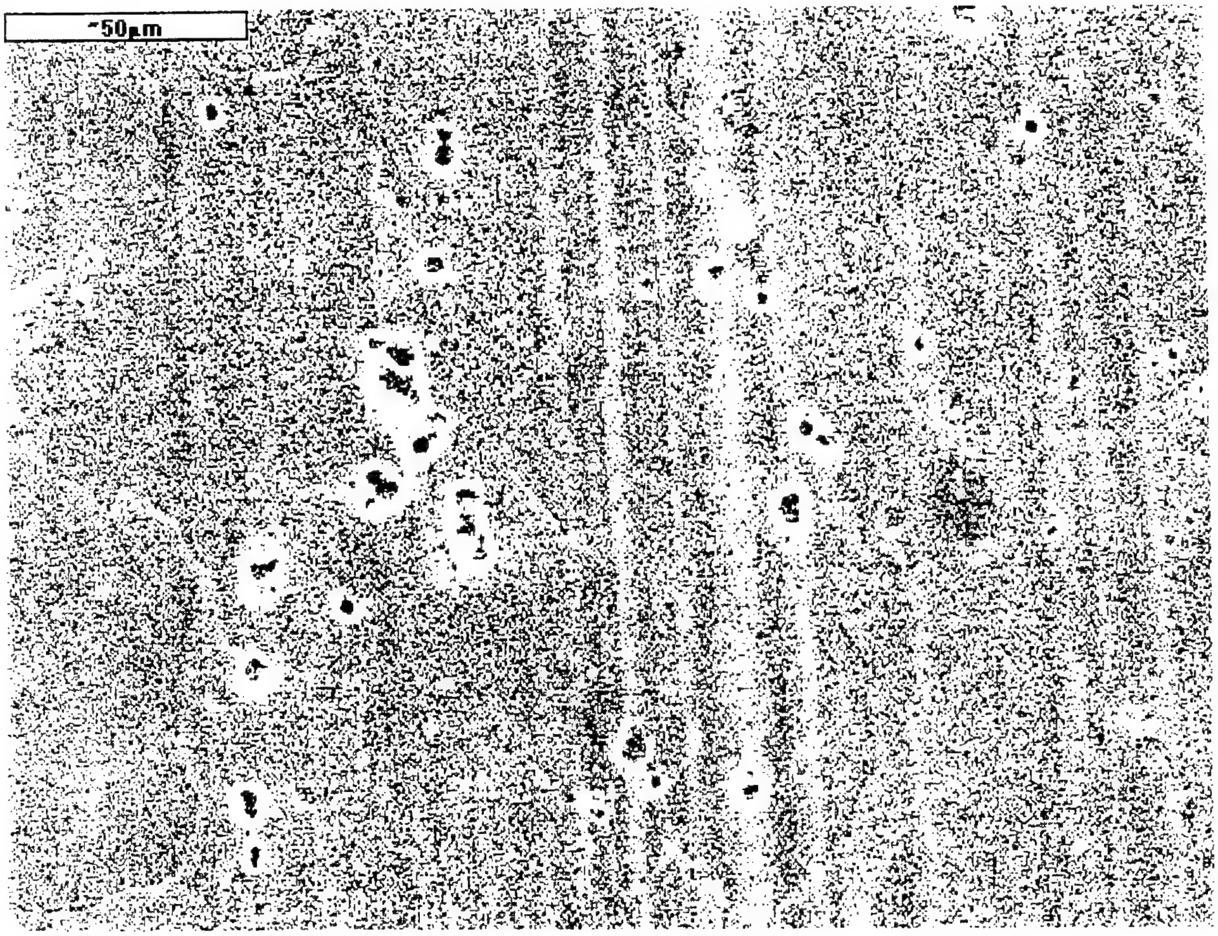


Fig. 10. Surface structure of Al 2024 T3 after two stage treatment. 1-st stage - 30 % H_3PO_4 + 25 ml/l H_2O_2, 40°C, 5 min; 2-nd stage - 100 ml/l HNO_3 + 25 ml/l H_2O_2, 25°C, 20 sec. Magnification 500.

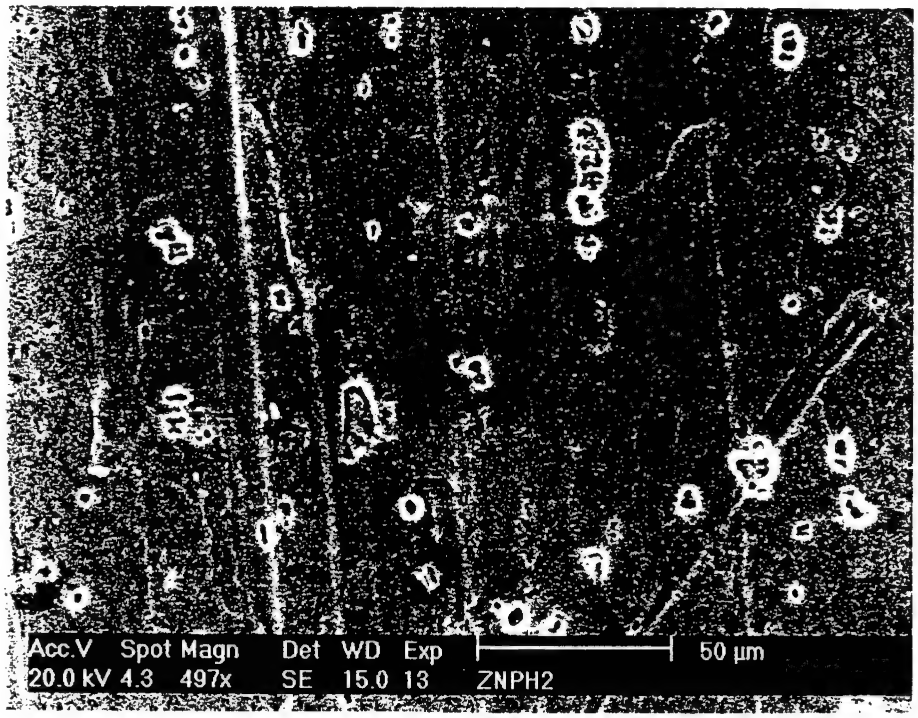


Fig. 11. Surface structure of Al 2024 T3 after treatment in solution containing 50ml/l HNO_3 , 50 ml/l HCl and 100 ml/l H_2O_2 at 25°C for 5 min. Magnification 500.

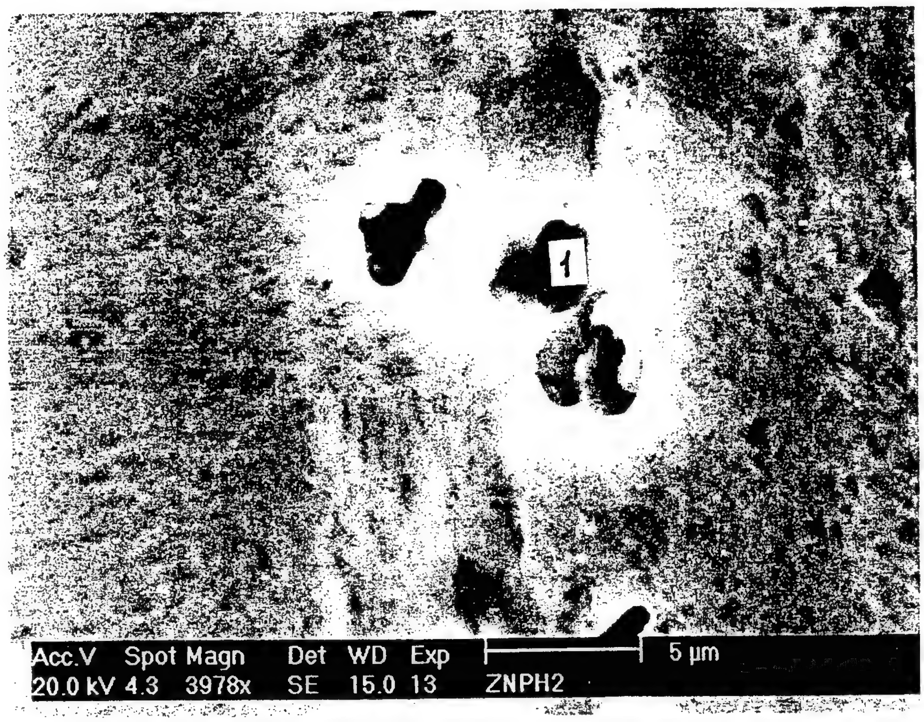


Fig. 12. Surface structure of Al 2024 T3 after treatment in solution containing 50ml/l HNO_3 , 50 ml/l HCl and 100 ml/l H_2O_2 at 25°C for 5 min. Magnification 4000.

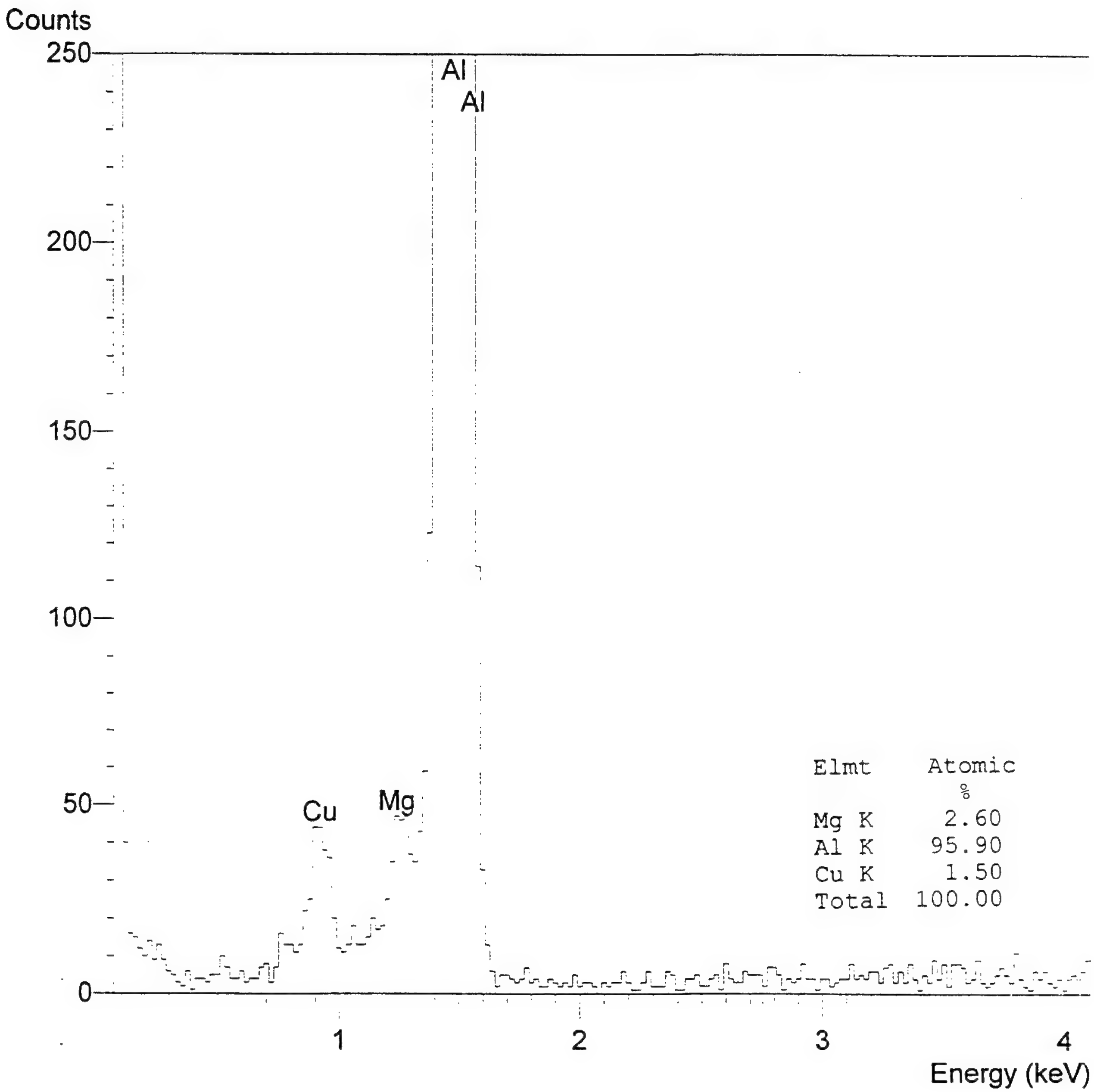


Fig. 13. EDS spectrum of Al 2024 T3 surface after treatment in a solution containing 50 ml/l HNO_3, 50 ml/l HCl and 100 ml/l H_2O_2. Area signed 1 in Fig. 12

3.3. Pretreatment in Bonder.

Several samples of Al 2024 T3 were treated in 30 % H_3PO_4 at 25 °C for 3 min, rinsed in water and immersed in a 3 % colloidal solution of SiO_2 at 25°C for 3 min. After that the samples were immersed in the phosphating solution and treated as usual. As can be seen in Fig. 14 the treatment in the colloidal SiO_2 solution contributed to the formation of faceted crystals.

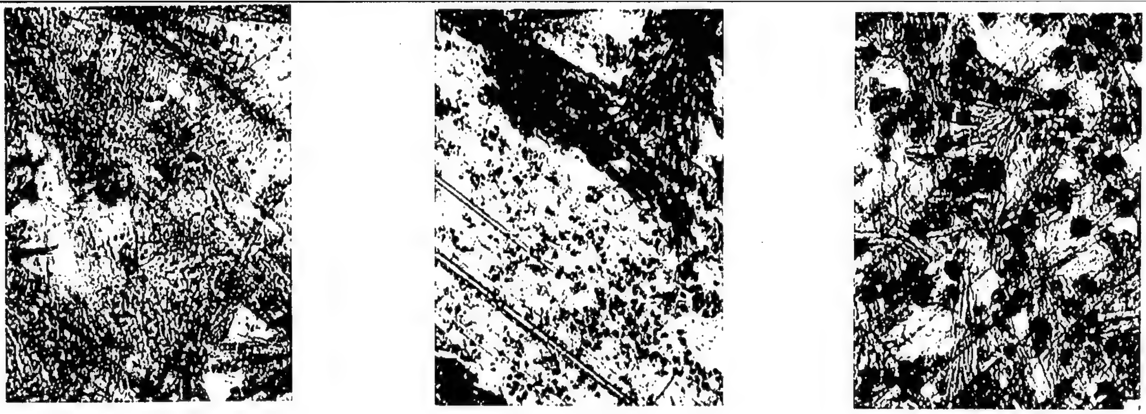
The samples obtained in this treatment were characterized by high corrosion resistance. They withstood 312 hour salt spray test but failed in Dry Tape Test and Scribed Tape Test. When the samples were pretreated in the SiO_2 solution only (without treatment in H_3PO_4 solution), nonuniform zinc/phosphate coatings were deposited. Examination of their paintability showed unsatisfactory adhesion to an Al 2024 T3 surface.

3.4. Pretreatment in alkaline solution.

Several Al 2024 T3 samples were treated in the solution containing 50 g/l NaOH at 25 °C for 1.5 min. The treatment resulted in the formation of a black film which could be removed by dipping in the solution containing 100 ml/l HNO_3 at 25°C for 1.5 min. However the surface was found to be very nonuniform. The increase of the treatment duration (in NaOH) up to 5 min led to even higher nonuniformity of the surface. The obtained black film could not be removed by treatment in HNO_3. The phosphating process was inhibited to a marked extent on the surfaces prepared in such way. The star like structure of the zinc / phosphate coating was weakly defined under the layer of small faceted crystals. Uncovered areas of Al 2024 T3 substrate could be seen (Fig. 15 ,sample 307). In contrast to this the star like structure of a zinc / phosphate coating deposited after pretreatment in H3PO4 was well defined. (Fig. 15, sample 308)

3.5. Pretreatment with sand blasting.

Several Al 2024 samples were subjected to sand blasting and then treated as shown in Table 6. Sand blasting essentially increased roughness of the surface . The surface structure before and after sand blasting is shown in Fig. 16. The immersion of sand blasted samples in H_3PO_4 solutions resulted in very intensive etching process with an extremely

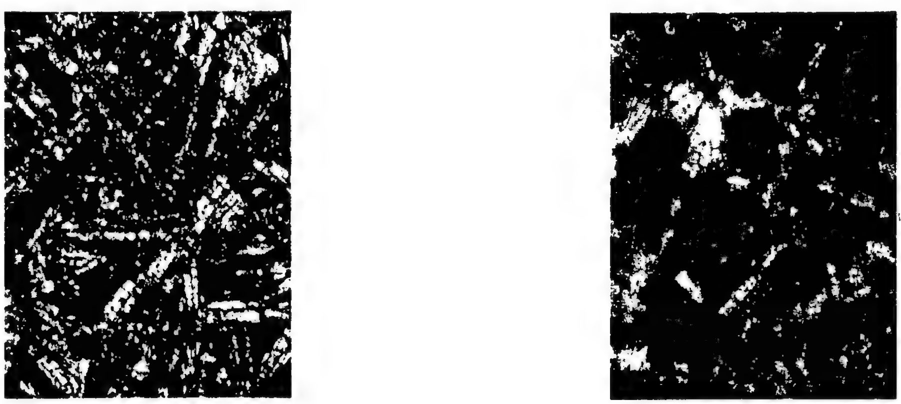


Without any pretreatment

Pretreatment in 3 % SiO_2

Pretreatment in
1) 30 % H_3PO_4
2) 3% SiO_2

Fig. 14. Initial stage of zinc / phosphate coating growth. Deposition time 2 min. Optical microphotographs. Magnification 200.



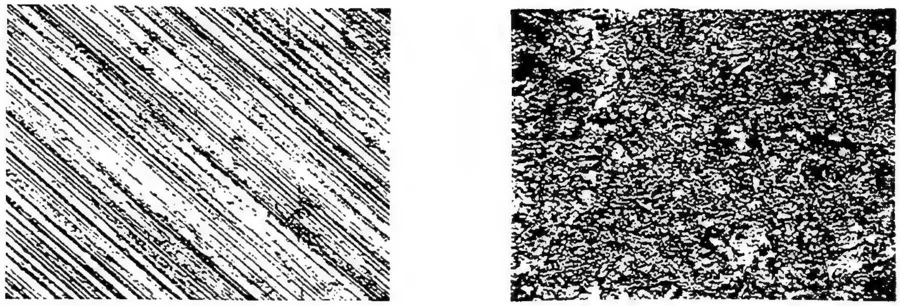
Pretreatment in 30 % H_3PO_4 at 25°C for 3 min
Sample 308

Pretreatment in
1) 50 g/l NaOH at 25°C for 5 min
2) 100 ml/l HNO_3 at 25°C for 1.5 min
Sample 307

Fig. 15. Effect of pretreatment on the structure of zinc / phosphate coating. Optical microphotographs. Magnification 200.

Table 6. Effect of pretreatment on corrosion resistance and paintability of samples preliminary subjected to sand blasting.

No	Pretreatment						Corrosion resistance (312 hrs. salt spray test)	Paintability (Dry Tape Test and Scribed Tape Test)
	1st stage			2nd stage				
	Solution comp.	T, °C	D, min	Solution comp.	T, °C	D, sec		
305	30% H_3PO_4	40	6	-	-	-		Did not meet the requirements but on the verge
337	-	-	-	-	-	-		Met the requirements
338	-	-	-	-	-	-	2 corr. stains after 144 hrs. 8 corr. stains after 312 hrs.	
339	30% H_3PO_4	25	3	-	-	-	No corrosion	
340	30% H_3PO_4	25	3	-	-	-		Met the requirements
341	30% H_3PO_4	25	3	100ml/l HNO_3	40	20		Met the requirements
344	30% H_3PO_4	40	5	100ml/l HNO_3				Did not meet the
345	+25ml/l H_2O_2	40	2.5	25ml/l H_2O_2	40	20		requirements but on the verge
350	50 ml/l HNO_3 +50 ml/l HCl	25	5	-	-	-		Did not meet the requirements but on the verge
351	+100 ml/l H_2O_2						2 corr. stains after 312 hrs.	
356	30% H_3PO_4	25	1.5	3 % SiO_2	25	180		Did not meet the requirements but on the verge
357							2 corr. stains after 192 hrs. 3 corr. stains after 312 hrs.	



a	б

Fig.16. Structure of Al 2024 T3 surface before (a) and (b) after sand blasting. Optical microphotographs. Magnification 200.

vigorous hydrogen evolution because of an increase of surface area of samples. Therefore, along with etching at 40°C for 6 min, etching under considerably mild conditions - 25 °C and 1.5 - 2.5 min, was carried out.

As could be seen from Table 6, these conditions provided better adhesion of a zinc/phosphate coating to a Al 2024 T3 surface. Deep etching resulted ,probably, in the formation of a surface film consisted primarily of particles (composition of particle is shown in Fig.3) unlinked with the surface (see Paragraph 3.1.). Optimal etching, in contrast, provided the formation of protrusions which gave a better adhesionadhesion. The fact that the sample that was not subjected to etching exhibited satisfactory paintability suggests a mechanical lock mechanism of adhesion. The treatment of sand blasted samples in acidic solutions of H_2O_2 did not provide reproducible results. The corrosion resistance of tested samples was satisfactory with the exception of sample 338 which was not treated chemically.

3.6. Composition of zinc phosphate coatings.

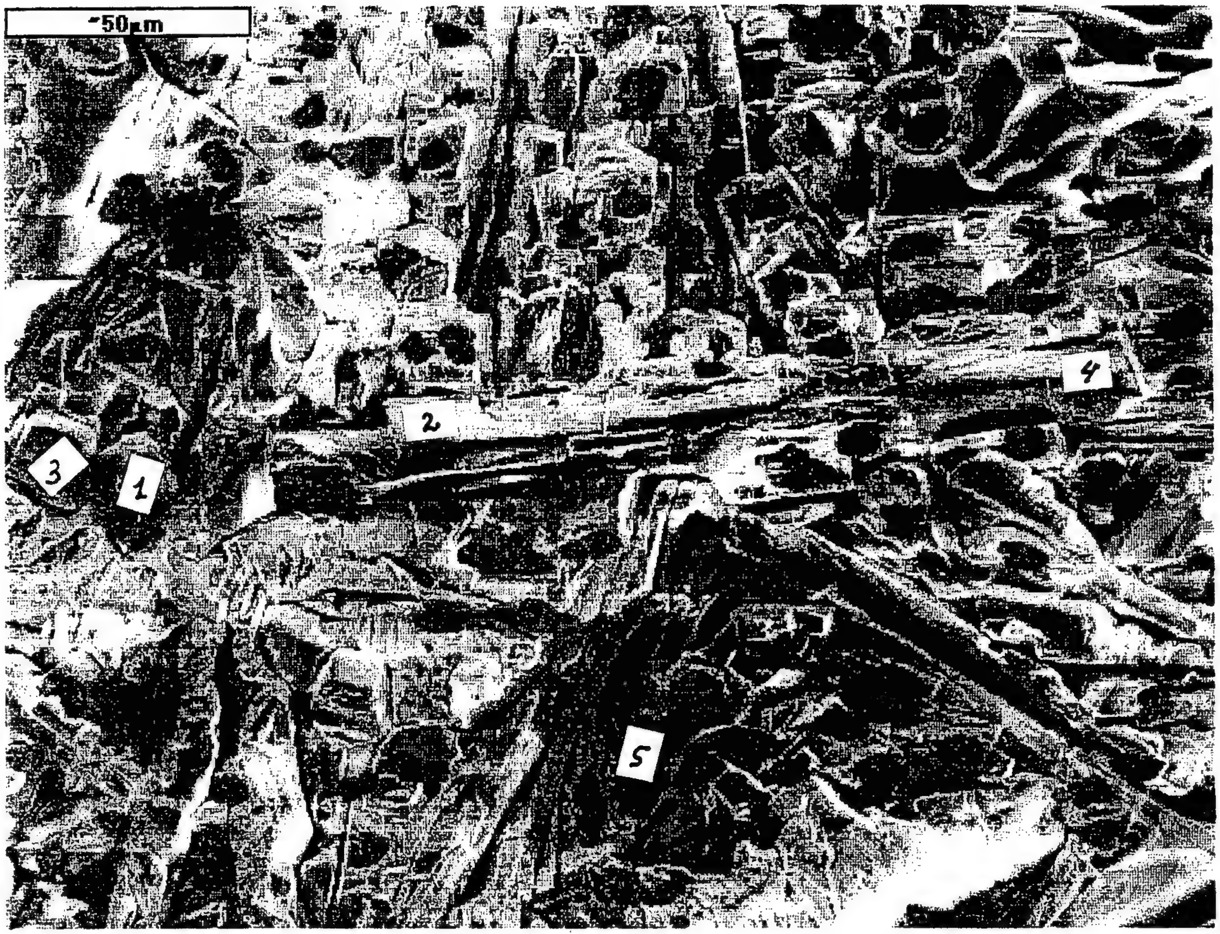
The composition the coatings was determined by EDS and XR analyses. The results of EDS analysis for two different samples are shown in Figs. 18,17. The crystals of different forms, namely, faceted and dendritic (star rays) had the same composition . The crystals consisted of Zn, P and O. The concentrations of these elements were about 22, 16 and 62 % (a.c.), respectively.

The results of XRD analysis for a Al 2024 T3 substrate and coating deposited on it (sample 240) are shown in Appendix B. The XRD analysis indicated of the presence of Zinc Phosphate Hydrate (Hopeite) with composition $Zn_3(PO_4)_2 \times 4 H_2O$ (Appendix C).

Zn / P/ O ratios in Hopeite were 1.5 and 6.0, respectively. However, according to the EDS analysis these ratios ranged between 1.42 / 1 / 3.94 and 1.73 / 1 / 5.49. The reasons of the discrepancy in the results of EDS and XRD analyses should be investigated at the following stage of the work.

Spot	Zn / P/ O
1	1.42 / 1 / 3.94
2	1.57 / 1 / 4.92
3	1.71 / 1 / 4.73

Fig.17. Surface morphologyand composition of zinc/phosphate coating. Sample 234.. Magnification 500.



Spot	Zn / P / O
1	1.73 / 1 / 5.49
2	1.72 / 1 / 5.27
3	1.50 / 1 / 4.28
4	1.61 / 1 / 4.88
5	1.56/ 1 / 4.41

Fig.18. Surface morphology and composition of zinc/phosphate coating. Sample 240. Magnification 500.

4. Summary.

* The structure, corrosion resistance and paintability of zinc / phosphate coatings on Al 2024 T3 were investigated in dependence on pretreatment conditions. The mechanical pretreatment of a substrate by sand blasting and chemical pretreatment in acid, alkaline and colloidal SiO_2 solutions were studied.

* Pretreatment in NaOH and colloidal SiO_2 solutions resulted in high nonuniformity of the coating thickness.

* The pretreatment in the solution of H_3PO_4 led to the formation of uniform zinc / phosphate coatings having high corrosion resistance. The coated samples withstood 312 hour salt spray test. The coatings had good adhesion to an epoxy primer ,but their adhesion to Al 2024 T3 surfaces was insufficient and did not meet the requirements of IAI PS 83.01 standard .

* The pretreatment in H_3PO_4 solutions resulted in the formation of a powdery film of corrosion products on Al 2024 T3 surface. This film contained cathodic zones of about 20% Cu. The removal of this film from the surface of Al 2024 T3 did not improved the paintability and decreased the corrosion resistance of the samples.

* The pretreatment in acidic solutions of H_2O_2 did not provide good reproducible results of the corrosion and paintability tests.

* The mechanical pretreatment in combination with mild etching in the solution of H_3PO_4 provided good results of the corrosion and paintability tests according to ASTM B 117 and IAI PS 83.01 standards, respectively.

*SEM and EDS analyses showed that Zinc / phosphate coatings consisted of star like and faceted crystals of the same composition, namely, %(a.c.): Zn - 28, P - 16, O - 62. According to the results of a XRD analysis the coating composition corresponded to Zinc Phosphate Hydrate (Hopeite) - $Zn_3(PO_4)_2 \times 4\, H_2O$.

*The mechanical pretreatment of a Al 2024 T3 substrate increased the surface roughness and allowed to produce samples which met the requirements of the paintability test. However, the corrosion resistance of these samples was unsatisfactory.

5. References.

1. Non-Chromium Coatings on Aluminum, Project No 524-767, USAF contact No F617088-97 WO111 by O. Berkh, G. Rogalsky, M. Rotel, J.Zahavi. Haifa, December 1997.

2. Non-Chromium Coatings on Aluminum, Special project No SPC-98-4005, USAF contact No F61775-98 WE 054 by O. Berkh, S. Tamir, A. Bodnevas , M. Rotel, J.Zahavi. Haifa,August, 1998.

Appendix A

IAI PS 83.01 standard.

7. EQUIPMENT

7.1 Stylus, metallic tip.

8. REQUIREMENTS

8.1 Perform the tests described in this PS after finishing, drying, or conditioning in accordance with the relevant finishing PS requiring adhesion testing.

8.2 The tape test location shall be in an area at least 6 mm away from rivets, screws, holes, edges and areas immediately surrounding ball or roller type bearings.

8.3 The number and exact location of the adhesion tape tests shall be determined by the Quality Control Department. If "wet" tape adhesion tests are required, the tests shall be performed only after examining the location by the "dry" method.

8.4 The scribed dry or wet tests shall be performed (per para. 9.3), only when specifically requested, on test specimens accompanying the parts during the finishing process.

9. PROCEDURES

9.1 Dry Tape Test Method

9.1.1 Clean area to be tested with a clean cheesecloth dampened with solvent. Wipe dry with clean cheesecloth before the solvent evaporates.

9.1.2 Apply a 15 cm long piece of masking tape (per para. 6.4) and firmly press it down with hand pressure or using the roll of masking tape itself.

9.1.3 Lift one end of the masking tape for a length of approximately 5 cm and remove the tape in one abrupt motion, pulling perpendicular to the test panel.

9.2 Wet Tape Test Method

9.2.1 Clean area to be tested with a clean cheesecloth dampened with solvent. Wipe dry with clean cheesecloth before the solvent evaporates.

9.2.2 Place a pad of cheesecloth, approximately 10 x 10 cm, that is saturated with distilled water, to the area to be tested. Cover the wet patch with a piece of polyethylene film and seal the edges with masking tape.

9.2.3 Allow the wet patch to remain on the coated surface for 24 hours and wetting the cheesecloth to saturation with distilled water as necessary.

9.2.4 After 24 hours, remove the wet pad and wipe the area with a clean cheesecloth.

9.2.5 Within 2 minutes firmly apply the masking tape and follow the procedure described in paras. 9.1.2 and 9.1.3.

9.3 Scribed Tape Test Method

9.3.1 With a stylus, scribe the test specimen to make two parallel scratches approximately 50 mm (2 in.) long and approximately 25 mm (1 in.) apart down the substrate. Join the parallel lines with an "X" across the center or by an inverted "V" at each end.

9.3.2 Apply the masking tape and follow the procedure described in paras. 9.1.2. and 9.1.3.

9.3.3 When performing a scribed "wet" tape adhesion test, wipe the surface with a clean cheesecloth and make the scratches within 2 minutes of the removal of the wet pad. Follow the procedure described in para. 9.2.5.

10. QUALITY ASSURANCE

10.1 General

The Quality Control inspection shall ensure the strict adherence to all requirements of this PS.

10.2 Coating Examination

10.2.1 After performing a tape adhesion test, examine the area for any coating damage. Removal of one or more of the coating layers from the substrate shall be considered as a failure of the paint to meet adhesion requirements. Use only masking tape per para. 6.4.

10.2.2 If the area of the coating failure is equal to or greater than 5-7 cm^2, the damage shall be considered as a "regional failure" and the coating shall be removed and the part refinished per the applicable PS.

10.2.3 If the area of the coating failure is less than 5-7 cm^2, perform three additional dry tape tests per para. 9.1, across the edges of the failed area.

10.2.4 Perform at least three additional dry type tests per para. 9.1 in areas surrounding the failure area in order to determine the maximal damaged area.

10.2.5 In case, no additional coating layers were removed during the additional tests per paras. 10.2.3 and 10.2.4 or the failure area is less than 5-7 cm^2 the damage shall be considered as a "local failure" and only a touch-up per the applicable PS is necessary.

10.2.6 In case, more additional coating layers were removed during the additional adhesion tests performed per paras. 10.2.3 and 10.2.4, and the failure area is equal to or greater than 5-7 cm^2 the damage shall be considered as a "regional failure" and the coating shall be removed and the part refinished per the applicable PS.

10.3 Records

10.3.1 Records of inspection and product testing shall be retained for a period specified in the relevant Divisional Quality Assurance Procedures.

10.3.2 The records shall contain the following minimum information:

a. Type of tape test method performed.
b. Test results.
c. Inspector's disposition including signature and QA stamp.
d. Part or assembly number.

Appendix B

XRD Pattern for sample 240

X'Pert Graphics & Identify | **mtrchu**
(searched) peak list: ol - 2 | **Date of edition: 1/13/99 18:33**

Description:

ol - 2

Original scan: ol - 2
Scan created: 1/12/99 12:40
Description of scan:
ol - 2

Peak search parameter set: As Measured Intensities
Set created: 5/6/98 11:20
Peak positions defined by: Minimum of 2nd derivative
Minimum peak tip width (°2The0.00
Maximum peak tip width (°2Th9.50
Peak base width (°2Theta): 20.00
Minimum significance: 1.00

d-spaci (nm)	Relative Intensity (%)	Angle (°2Theta)	Peak Height (cps)	Backgroun (cps)	Tip Width (°2Theta)	Significanc
9.1430	81.65	9.6655	6234	42	0.0900	10.64
7.5429	2.30	11.7225	175	42	0.9600	1.27
5.3045	5.26	16.6991	402	42	0.0900	2.18
5.0924	8.18	17.3997	625	42	0.1200	6.63
4.8526	2.56	18.2670	196	42	0.0900	1.52
4.5734	100.00	19.3926	7635	42	0.1200	24.22
4.4085	4.54	20.1254	347	42	0.0900	1.98
4.0660	0.73	21.8403	56	42	0.0900	2.10
4.0009	8.93	22.2001	682	42	0.1200	6.77
3.8793	2.34	22.9051	178	42	0.0900	1.49
3.6451	1.85	24.3991	141	42	0.1200	2.53
3.4612	24.43	25.7170	1865	42	0.1200	10.91
3.3880	12.99	26.2823	992	42	0.1200	7.55

d-spaci (nm)	Relative Intensity (%)	Angle (°2Theta)	Peak Height (cps)	Backgroun (cps)	Tip Width (°2Theta)	Significanc
3.1299	2.13	28.4934	162	42	0.0900	1.02
3.0473	1.09	29.2828	83	42	0.0900	3.52
3.0122	1.88	29.6321	143	42	0.1200	1.92
2.9576	1.13	30.1922	86	42	0.1200	1.71
2.8526	23.53	31.3314	1797	42	0.1500	16.29
2.7588	0.91	32.4249	69	42	0.1200	1.00
2.6494	3.97	33.8031	303	42	0.1500	4.69
2.6124	6.94	34.2976	530	42	0.1500	6.96
2.5457	4.02	35.2249	307	42	0.1200	2.44
2.5164	1.50	35.6479	115	42	0.0900	3.10
2.4718	1.77	36.3143	135	42	0.1800	3.44
2.4248	2.00	37.0438	153	42	0.0900	6.40
2.3382	33.59	38.4674	2565	42	0.1800	18.48
2.3069	1.05	39.0110	80	42	0.1800	1.02
2.2866	13.83	39.3707	1056	42	0.1200	3.82
2.2695	2.16	39.6795	165	42	0.1500	1.60
2.1885	0.83	41.2145	63	42	0.0900	1.38
2.1506	0.47	41.9752	36	42	0.3600	3.72
2.0988	4.39	43.0615	335	42	0.0900	1.38
2.0923	4.35	43.2011	332	42	0.0900	2.62
2.0262	27.61	44.6858	2108	42	0.2400	29.29
2.0007	3.69	45.2880	281	42	0.0900	1.04
1.9735	0.52	45.9478	40	42	0.1800	1.25
1.9381	5.05	46.8354	386	42	0.1200	3.02
1.9039	0.23	47.7289	18	42	0.2400	1.14
1.8591	0.39	48.9516	30	42	0.2400	1.11
1.8250	1.79	49.9286	137	42	0.3000	5.02
1.7761	0.63	51.4028	48	42	0.0900	1.25
1.7355	0.93	52.6978	71	42	0.0900	1.39
1.7298	0.91	52.8822	69	42	0.0900	1.72
1.6950	1.02	54.0558	78	42	0.1200	1.10
1.6634	0.90	55.1688	69	42	0.1800	2.35

d-spaci (nm)	Relative Intensity (%)	Angle (°2Theta)	Peak Height (cps)	Backgroun (cps)	Tip Width (°2Theta)	Significanc
1.6362	1.09	56.1675	84	42	0.0900	1.14
1.6129	0.34	57.0535	26	42	0.3000	1.40
1.5947	0.44	57.7661	33	42	0.2400	1.57
1.5665	2.18	58.9036	166	42	0.0900	1.07
1.5246	11.81	60.6895	902	42	0.0900	3.91
1.5204	5.23	60.8750	400	42	0.0600	18.17
1.5053	1.16	61.5522	88	42	0.0900	1.30
1.4320	40.98	65.0799	3129	42	0.1500	10.20
1.4277	19.38	65.3014	1480	42	0.0900	9.59
1.3465	0.56	69.7875	43	42	0.2400	1.07
1.3220	1.12	71.2718	85	42	0.0900	3.48
1.3187	0.69	71.4813	53	42	0.1200	1.28
1.3042	0.19	72.3993	15	42	0.4800	1.01
1.2694	0.91	74.7148	69	42	0.1200	1.22
1.2302	1.16	77.5253	89	42	0.0600	2.94
1.2215	6.97	78.1885	532	42	0.2400	8.17
1.2179	3.83	78.4628	292	42	0.1200	1.09
1.1694	2.26	82.3973	172	42	0.1500	1.61
1.0911	0.72	89.8062	55	42	0.1200	1.13
1.0487	0.11	94.5291	8	42	0.7200	1.50
1.0134	1.12	98.9437	85	42	0.4200	3.69
0.9291	1.79	112.0033	137	42	0.4800	5.64
0.9057	2.47	116.5230	189	42	0.2400	2.11

XRD pattern for AL 2024 T3 substrate

X'Pert Graphics & Identify **mtrchu**
(searched) peak list: ol - 1 **Date of edition: 1/12/99 12:41**

Description:

ol - 1

Original scan: ol - 1
Scan created: 1/12/99 11:22
Description of scan:
ol - 1

Peak search parameter set: As Measured Intensities
Set created: 5/6/98 11:20
Peak positions defined by: Minimum of 2nd derivative
Minimum peak tip width (°2The0.00
Maximum peak tip width (°2Th9.50
Peak base width (°2Theta): 20.00
Minimum significance: 1.00

d-spaci (nm)	Relative Intensity (%)	Angle (°2Theta)	Peak Height (cps)	Backgroun (cps)	Tip Width (°2Theta)	Significanc
7.2944	2.85	12.1233	159	49	2.4000	11.60
3.6504	0.15	24.3632	8	49	2.8800	1.18
2.8211	0.35	31.6904	20	49	0.1500	1.06
2.5862	0.69	34.6551	39	49	0.1800	1.00
2.5541	0.72	35.1054	40	49	0.1800	1.58
2.3358	100.00	38.5094	5567	49	0.2100	38.98
2.2467	0.99	40.1000	55	49	0.3000	1.44
2.0977	1.59	43.0857	89	49	0.2100	1.78
2.0247	96.17	44.7210	5353	49	0.2700	58.66
1.5842	0.54	58.1847	30	49	0.1800	1.91
1.4311	86.41	65.1232	4810	49	0.1500	11.67
1.4272	43.66	65.3283	2430	49	0.0900	1.46
1.2209	13.65	78.2341	760	49	0.2100	6.39

X'Pert Graphics & Identify **mtrchu**
(searched) peak list: o! - 1 **Date of edition: 1/12/99 12:41**

d-spaci (nm)	Relative Intensity (%)	Angle (°2Theta)	Peak Height (cps)	Backgroun (cps)	Tip Width (°2Theta)	Significanc
1.2178	8.61	78.4667	479	49	0.0900	1.64
1.1685	4.41	82.4781	245	49	0.1200	1.02
1.0129	2.46	99.0080	137	49	0.3000	2.01
0.9290	3.18	112.0048	177	49	0.4200	4.23
0.9055	5.29	116.5542	294	49	0.2400	1.46

Appendix C

Reference pattern for Hopeite $Zn_3(PO_4)_2 \times 4\ H_2O$

X'Pert Graphics & Identify **mtrchu**

Date of edition: 1/13/99 18:43

Reference pattern: 37-0465 Reference database: c:\identdb

PDF Name: Zinc Phosphate Hydrate
Mineral Name: Hopeite
Chemical Name: -
Alternate Name: -

Formula: Zn3(PO4)2!4H2O

Elements:

Chemical groups:

Subfiles:

Radiation: Cu K-Alpha1
Wavelength (nm): 0.154056

Peaks

d-spacing (nm)	Relative Intensity (%)	Angle (°2Theta)
0.912000	60.00	9.6900
0.529700	12.00	16.7230
0.509200	16.00	17.4014
0.484900	18.00	18.2807
0.456800	100.00	19.4158
0.456800	100.00	19.4158
0.441000	20.00	20.1185
0.407000	4.00	21.8190
0.400200	18.00	22.1944
0.388000	10.00	22.9015
0.364600	6.00	24.3932
0.364600	6.00	24.3932
0.346000	25.00	25.7264
0.338800	25.00	26.2828
0.322500	1.00	27.6369

d-spacing (nm)	Relative Intensity (%)	Angle (°2Theta)
0.313200	6.00	28.4746
0.305000	1.00	29.2570
0.301200	4.00	29.6345
0.295900	7.00	30.1778
0.285300	65.00	31.3273
0.285300	65.00	31.3273
0.275700	2.00	32.4477
0.265100	13.00	33.7832
0.261200	16.00	34.3031
0.258400	3.00	34.6866
0.254700	10.00	35.2068
0.253300	7.00	35.4078
0.251500	9.00	35.6697
0.244700	1.00	36.6957
0.244700	1.00	36.6957
0.242400	6.00	37.0565
0.234200	5.00	38.4039
0.231900	1.00	38.7999
0.228700	10.00	39.3650
0.226800	8.00	39.7086
0.226800	8.00	39.7086
0.225400	2.00	39.9657
0.220500	3.00	40.8931
0.218900	1.00	41.2055
0.215700	4.00	41.8453
0.214700	1.00	42.0494
0.212800	1.00	42.4429
0.210000	9.00	43.0368
0.210000	9.00	43.0368
0.203600	1.00	44.4606
0.200200	9.00	45.2571
0.200200	9.00	45.2571
0.197500	1.00	45.9109

d-spacing (nm)	Relative Intensity (%)	Angle (°2Theta)
0.152990	4.00	60.4615
0.152550	15.00	60.6542
0.151500	1.00	61.1194
0.151500	1.00	61.1194
0.150870	4.00	61.4021
0.150580	4.00	61.5332
0.149450	1.00	62.0497
0.147990	2.00	62.7309
0.146420	1.00	63.4814
0.146420	1.00	63.4814
0.145500	1.00	63.9301
0.145080	1.00	64.1373
0.144320	3.00	64.5157
0.143770	1.00	64.7926
0.143180	1.00	65.0925
0.142610	2.00	65.3850
0.141640	2.00	65.8894
0.141640	2.00	65.8894
0.141250	1.00	66.0946
0.140720	2.00	66.3756
0.140040	1.00	66.7399
0.137850	1.00	67.9431
0.136490	1.00	68.7142
0.136050	1.00	68.9677
0.135610	2.00	69.2233
0.135610	2.00	69.2233
0.134750	2.00	69.7288
0.134470	1.00	69.8951
0.133460	1.00	70.5023
0.132570	1.00	71.0469
0.132200	2.00	71.2761
0.132200	2.00	71.2761
0.131030	1.00	72.0113

d-spacing (nm)	Relative Intensity (%)	Angle (°2Theta)
0.130410	1.00	72.4077
0.129400	1.00	73.0638
0.129400	1.00	73.0638
0.128200	1.00	73.8605
0.127860	1.00	74.0897
0.127860	1.00	74.0897
0.127370	1.00	74.4228
0.126950	1.00	74.7110
0.126540	1.00	74.9947
0.126540	1.00	74.9947
0.125610	1.00	75.6470
0.125610	1.00	75.6470
0.124590	1.00	76.3772
0.123690	1.00	77.0345
0.123060	3.00	77.5022
0.121360	1.00	78.7965
0.121360	1.00	78.7965
0.120620	1.00	79.3751
0.119660	1.00	80.1401
0.119270	1.00	80.4557
0.118430	1.00	81.1450
0.118080	1.00	81.4361
0.118080	1.00	81.4361
0.117280	1.00	82.1106
0.117280	1.00	82.1106
0.117020	1.00	82.3325
0.116330	1.00	82.9282
0.115990	1.00	83.2254
0.115680	1.00	83.4984
0.115180	1.00	83.9432
0.114730	1.00	84.3481
0.114730	1.00	84.3481
0.114380	1.00	84.6662